Analysis of Jet Fire on Intumescent Coating Applied on Steel Substrate

by

Ahmad Fairus Bin Zamri

Dissertation submitted in partial fulfillment of
the requirements for the
Bachelor of Engineering (Hons)
(Mechanical Engineering)

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CERTIFICATION OF APPROVAL

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A project dissertation submitted to the
Mechanical Engineering Programme
Universiti Teknologi PETRONAS
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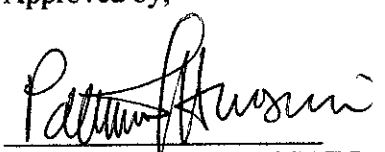
Approved by,



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CERTIFICATION OF ORIGINALITY

This is to certify that I am responsible for the work submitted in this project, that the original work is my own except as specified in the references and acknowledgements, and that the original work contained herein have not been undertaken or done by unspecified sources or persons.

AHMAD FAIRUS BIN ZAMRI

ABSTRACT

The use of intumescent coatings is one of the easiest, economical and most efficient ways to protect steel component from heat generated in fire. By insulated the steel substrates, the rate of temperature increase experienced by component from fire are substantially reduced. Thus, this insulation imparts additional time for the application of fire suppression and cooling the coated steel component reduces the potential for a material failure of the component or structure. The purpose of this report written was to discuss the progress job done; basic understanding, methodology and result obtain regarding the final year project about the analysis of jet fire on intumescent coating applied on steel substrate. The main objective of this project was to investigate and study the interface bonding between intumescent and the steel substrate before and after the jet fire burning. The investigation revealed the achievement performance intumescent coating as fire retardant. The project was focused on the durability of intumescent coating applied on the steel. The project also includes the characteristic study of the intumescent material, the coating method that been applied on the steel, and also to investigate the interface bonding between the intumescent and steel, before and after exposed to the heat.

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CHAPTER 1
INTRODUCTION

1.1 BACKGROUND STUDIES

Oil, gas and petrochemical industries operation that involve the flammable material in their basic operation can be the most dangerous workplace to the workers. Thus, highest priority has been given to all kind of operation in oil and gas industries that involves potential fire hazard such as implementing the fire protection system and emergency respond system.

From the transportation to processing and storage area, the potential for fire to occur is very high. In order to reduce the risk of this accident, most of the oil and gas companies have implemented and developed a Fire Protection and Control solutions for continuity of their operations with low risk in stake.

Meanwhile, in the construction and facilities industries, most of structure, tool and facilities are made from steel. According to studies, steel is very sensitive material when exposed to the heat, by losing its mechanical properties; the strength, ductility, and atomic structure. The uncoated steel structure cannot withstand at very high temperature, especially the temperature due to the outside explosion or fire. The potential of steel type structures to collapse is very high if these structures being exposed to excessive heat beyond the temperature of steel can sustain before its physical characteristic changed. The massive heat receive by the steel will result the steel to elongate and expanding further from its original shape. The sudden changes in length will cause the defect to structure and indirectly lead to the leakage or structure collapse.

This phenomenon may cause a structure failure especially for the steel pipes, steel vessel and highly sealed steel structure.

In the processing industries especially oil and gas, steel pipes are widely used as the main transportation system to transfer oil or gas from one station to another. This pipelines lie buried for hundreds of years underground or lie outside surrounding depending on the distance and location of pipeline built. With the same characteristic of the steel properties, this steel pipes will lose it mechanical properties when it expose to the high temperature surrounding such as the strength, ductility and also the structure. As the temperature increase, the steel pipes tend to extent, the sudden temperature rising will indirectly initiate leaking on the steel pipes structure.

Many of the incidents related to the explosion of the pipeline and also fire in oil, gas and petrochemical industries has been reported, and most of it consumes human life. In 13 December 2000, at least six people have been killed and several others injured in an explosion along Ecuador's only crude oil pipeline [1]. The latest accident has been reported on 30 November 2007, where two workers killed in an explosion of major oil pipeline in Minnesota [2].

Today, the intumescent coating layer has been implemented widely in structure as well as highly sealed steel structure as fire protection mechanism. As passive fire-retardant, intumescent has being permanently attached to the material as coating layer for a longer period of time and once activated when the fire or heat existed. Intumescent coating will delay the heat transfer to the structure inside by forming protecting layer.

1.2 PROBLEM STATEMENT

The use of intumescent coatings is one of the easiest, economical and most efficient ways to protect components from heat generated in fire. By insulated these

components, the rate of temperature increase experienced by these components from fire are substantially reduced. Thus, this insulation imparts additional time for the application of fire suppression and cooling the coated component reduces the potential for a material failure of the component.

These fires retardant coating has been particularly useful on ship, oil platforms, and other hydrocarbon storage and processing facilities. Although the coating material applied is proved as the best heat protection material, the ineffective coating practiced will reduce the durability of these coating or vice versa.

It is necessary to study the intumescent fire resistive coatings which are designed to perform under severe conditions and maintain the steel integrity between one and three hour when the temperature of the surroundings is in excess of 1100 °C. Hence it can be applied to the oil and gas industry field. In order to prove that, interface studies on the bonding between the intumescent, primer and steel substrate before and after exposed to heat are importance in order to determine the durability of the coating as fire protection system.

1.3 OBJECTIVE AND SCOPE OF STUDY

The main objectives of this project are follows:

1. To investigate the interface integrity between intumescent and steel substrate before and after jet fire;
2. To study and understand the characteristic of the epoxy intumescent material;
3. To identify, study and understand about the available coating process that can be applied for intumescent to the steel substrate;
4. To perform the analysis on interface bonding between intumescent material and steel before and after the jet fire burning test; and

5. To identify the changes occur on the coating and steel substrate after the jet fire.

The scope of work for this project was basically analysis on the effect of the reactive epoxy intumescent towards the bonding and also the steel substrate, and coating effectiveness. The analysis works were focused more on the characteristic changes of the coating and steel substrate due to jet fire. Several tests were carried out to determine the performance of coating and also the intumescent characteristic. The results acquired from the investigation and researches were analyzed to determine any changes occurred on bonding between the intumescent and steel substrate due to jet fire. The investigation field of this project been expanded further by focusing on the steel microstructure before and after the jet fire burning test for coating performance evaluations. Besides analyzed the coating durability, the project also gives several attentions on the method of applying the coating on the steel surface.

CHAPTER 2
LITERATURE REVIEW

2.1 INTUMESCENT

Intumescence is defined as the swelling of certain substances when they are heated. Intumescent technologies have been accepted as one of passive fire-retardant material in passive fire protection systems, which help in delaying or preventing the combustion or flame from spreading further. Intumescent can be categories as chemical type fire retardant material, which initiate a chemical reaction by forming an insulative barrier between the fire and substrate.

As passive fire-retardant material, intumescent has being permanently attached to the material surface for a longer period of time and once activated when the fire or heat existed.

2.1.1 Advantages of intumescent as fire-retardant coating

The use of intumescent fire-retardant coating is one of the easiest, economical and the most efficient ways to protect materials against fire [3]. Intumescent coatings present three main advantages such as;

- It can prevent heat from penetrating and flames from spreading;
- it does not modify the intrinsic properties of the materials (e.g. the mechanical properties) ; and

- it is easily processed and may be used onto several materials such as metallic materials, polymers, textiles and wood.

2.1.2 Reaction towards fire

Intumescent coatings form on heating an expanded multicellular layer, which acts as an insulative thermal barrier which effectively protects the substrate against rapid increase of temperature, thereby maintaining the structural integrity of the substrate. Intumescent coatings contain "active" ingredients bound together by a binder. Generally, three "active" ingredients are used: an acid source (normally ammonium polyphosphate or a mineral acid), a carbon source (such as char forming polymers or polyols) and a blowing agent. The formulation of the coating has to be optimized in terms of physical and chemical properties in order to form an effective protective char [4].

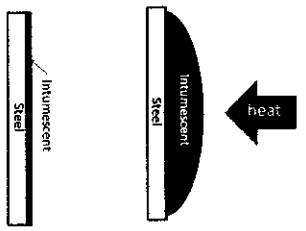


Figure 1: Reaction of intumescent material towards high temperature heat. (Before and after heat present)

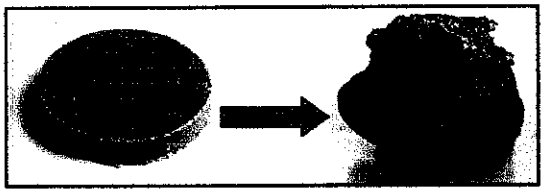


Figure 2: Swelling of intumescent coating. [7]

Chemical interactions between the "active" ingredients in the formulation lead to the formation of the intumescent char. It is generally accepted [5-6] that first, the acid source breaks down to yield a mineral acid, then it takes part in the dehydration of the carbonization source to yield the carbon char and finally, the blowing agent decomposes to yield gaseous products. The latter cause the char to swell and produce the insulating multi-cellular protective layer. This protective char limits both the heat transfer from the heat source to the substrate and the mass transfer from the substrate to the heat source, resulting in conservation of the underlying material.

2.1.3 Cooling effect

Typically, intumescent materials also contain a significant amount of hydrates. As the hydrates are spent, water vapor is released and produces a cooling effect. Once the water is spent, it is only the insulation characteristics of the char that was produced, which can slow down heat transfer from the exposed side to the unexposed side of substrate.

2.1.4 Durability of protection against fire

Typically, the expansion pressure that is created for intumescent is very low, because the soft carbonaceous char has little substance, which is beneficial if the aim is to produce a layer of insulation.

The duration of protection layer produce from the intumescent reaction with the heat are depends on the thickness of the layer applied. The thicker the layers the longer the protection layer withstand the heat. Intumescent start to foam when the surface

temperature reaches about 300°C by heating and form a layer of carbonaceous char 25 to 50 times thicker than at room temperature. As the thickness before intumescence is about 1 to 2 mm, intumescemt can be used for decorative purposes by the coating methods equivalent to those of ordinary coatings. For these advantages, intumescent coatings are used mainly in Europe as general fireproofing materials [9]. And these types of coatings are designed to perform under severe conditions and to maintain the steel integrity between one and three hour when the temperature of the surroundings is in excess of 1100 °C [10].

Figure 3: Brief Explanation on intumescent reaction. (Pipes covered with a thin-film intumescent spray fireproofing product called Unitherm. As the flame from the blow-torch hits it, the intumescent expands, forming a layer of insulation, which slows down heat transfer to the pipe below. Hydrates within the coating give up their water content, maintaining a temperature near the boiling point of 100 °C) [8].

2.1.5 Classifications of intumescents

Intumescent material can be classified into several categories such as epoxy and solvent-based, which based on their composition. Both type of intumescent have basic intumescent characteristic as written above but have different level of quality and performance.

2.1.6 Intumescent Coating on Metallic Substrate

The use of fire retardant coatings is one of the easiest, oldest most efficient ways to protect materials against fire. Indeed, fire retardant coatings presents several advantages such as, able to maintain the intrinsic properties of the materials like mechanical properties, easily processed and may be used onto several materials such as metallic materials, polymers, textiles and wood. The focusing on surface protection of the material is importance since the ignition usually occurs on the surface. The protection of metallic materials against the fire has become an important issue in the construction industry due to reason of materials distort leading to the collapse of building structures.

Intumescent paints which acting based on the intumescent technologies principles, prevents heat from penetrating and flames from spreading, by forming thermal barrier from expanded multicellular layer . As a consequence, this insulative barrier makes intumescent coatings particularly suitable for the protection of structural steelwork.

Intumescent paints are composed of three active ingredients; an acid source (generally ammonium polyphosphate—APP), a carbon source (such as (di)pentaerythritol) , and a blowing agent (most often melamine) linked together by a binder [15].

Several studies [16], [17], [18] have demonstrated that some polymers can participate in the intumescent phenomenon. If a binder acts in this way, it becomes a fundamental ingredient of the intumescent paint. As a consequence, this study will investigate the relationships between the polymer binder characteristics and the protective behavior of intumescent coatings. Linear and cross-linked substituted styrene/acrylic copolymers are studied.

2.2 HYDROCARBON FIRE CHARACTERISTIC

Hydrocarbon fires are characterized by a rapid and massive temperature increase as the fuel ignites usually occurs in oil and gas field. This type of fire also causes a high level of pressure that would seriously compromise normal thin-film intumescents. Most other traditional structural steelwork fire protection systems would also find it difficult to cope with this level of force.

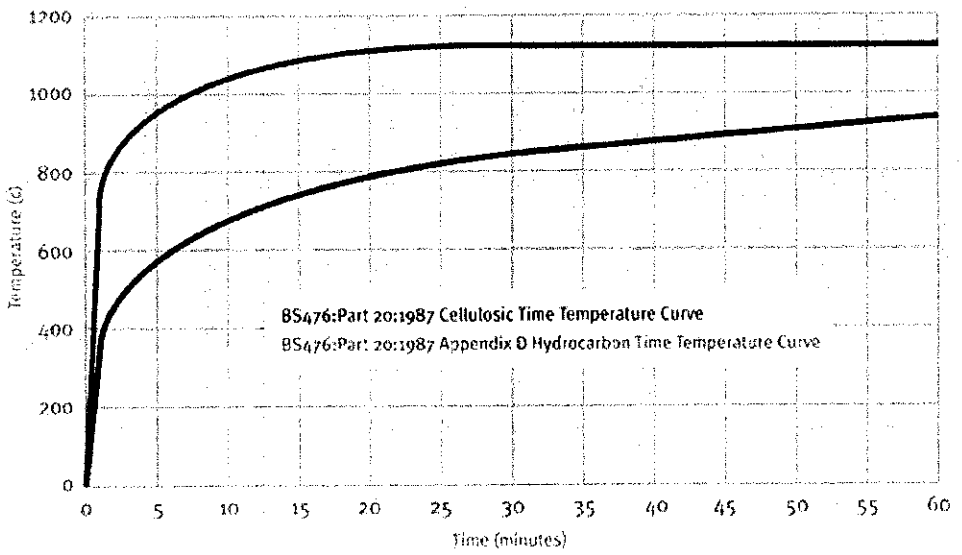


Figure 4: Time temperature curves for 'hydrocarbon' fire and 'cellulosic' fires [11]

The graph (Figure 4) demonstrates the different time temperature curves for this kind of 'hydrocarbon' fire and 'cellulosic' fires (as used for most Building Regulations standards on fire protection) [11]. The graph below interpret that the Hydrocarbon time temperature curve increase rapidly at the beginning of the graph and reach higher temperature compare to the Cellulosic time temperature curve ('cellulosic' fires curve).

2.3 LOW- CARBON STEEL CHARACTERISTIC

Low-carbon steels (AISI 1005-1026, 1108-1119, 1211-1215, and 1513-1527), by definition, contains less carbon (0.05–0.15% carbon content) than other steels and are inherently easier to cold-form due to their soft and ductile nature. When strength is not a major concern, low-carbon steels are good choices because they are easy to handle (draw, bend, punch, swage, etc.) and fairly inexpensive.

Carbon steels are steels whose alloying elements do not exceed the following limits:

TABLE 1: Carbon steels alloying elements [19]

ELEMENT	MAXIMUM WEIGHT %
C	1.00
Cu	*0.60*
Mn	*1.65*
P	*0.40*
Si	*0.60*
S	*0.05*

TABLE 2: General Properties of Steels at room temperature [20]

PROPERTIES	CARBON STEELS	ALLOY STEELS	STAINESS STEELS	TOOL STEELS
Density (1000kg/m^3)	7.85	7.85	7.75-8.1	7.72-8.0
Elastic Modulus (GPa)	190-210	190-210	190-210	190-210
Poisson's Ratio	0.27-0.3	0.27-.03	0.27-0.3	0.27-0.3
Thermal Expansion (10^{-6}/K)	11-16.6	9.0-15	9.0-20.7	9.4-15.1
Melting Point (°C)	-	-	1371-1454	-
Thermal Conductivity (W/m-K)	24.3-65.2	26-48.6	11.2-36.7	19.9-48.3
Specific Heat (J/kg-K)	450-2081	452-1499	420-500	
Electrical Resistivity (10^{-9}W-m)	130-1250	210-1251	75.7-1020	
Tensile Strength (MPa)	276-1882	758-1882	515-827	640-2000
Yield Strength (MPa)	186-758	366-1793	207-552	380-440
Percent Elongation (%)	10-32	4-31	12-40	5-25
Hardness (Brinell 3000kg)	86-388	149-627	137-595	210-620

The Table 2 lists the typical properties of steels at room temperature (25°C). The wide ranges of ultimate tensile strength, yield strength, and hardness are largely due to different heat treatment conditions.

2.4 ADHESION

Adhesion can be easily defined as the attraction between dissimilar bodies for one another. ASTM D 907 on Terminology of Adhesives addresses adhesion as "The state in which two surfaces are held together by interfacial forces which may consist of valence forces or interlocking action or both." In contrast to traditional joining methods such as riveting and screwing, bonding has no adverse effect on the material characteristics of the surfaces to be bonded such as drilling of holes in the joined parts, damaging and weakening them.

In the adhesion assessment, there have two different aspects involve:

1) Basic adhesion;

Signifies the summation of all interracial, intermolecular forces

2) Practical adhesion;

Used to represent the forces or work required to disrupt the adhering system [12].

2.5 THEORIES OF BASIC ADHESION

Bonding between polymeric coatings and substrates may be viewed as the union of two contiguous polymer phases, one a solid and the other a liquid which solidifies to form a thin film. The reversible separation of the two phases may be expressed by the work of adhesion

The bonding of an adhesive to an object or a surface is the sum of a number of mechanical, physical, and chemical forces that overlap and influence one another. As it is not possible to separate these forces from one another, we distinguish between mechanical interlocking, caused by the mechanical anchoring of the adhesive in the pores and the uneven parts of the surface, electrostatic forces, as regard to the difference in electronegativities of adhering materials, and the other adhesion mechanisms dealing with intermolecular and Chemical bonding forces that occur at the interfaces of heterogeneous systems.

This chemical adhesion mechanism is explained in the case of the intermolecular forces by the adsorption theory and in the case of chemical interactions by the chemisorption theory. The processes that play a role in the bonding of similar types of thermoplastic high-polymer materials, e.g. homogeneous systems, can be determined with the diffusion theory.

Most theories deal exclusively with the mechanisms of bond formation and disregard the fact that bond strength is ultimately a function of both the degree of bond formation, the nature of the bond (chemical and physical), and the rheological properties of the bonding phases. Summary paragraphs about basic theories of adhesion follow below.

2.5.1 Adsorption Theory

The adsorption theory states that adhesion results from intimate intermolecular contact between two materials, and involves surface forces that develop between the atoms in the two surfaces.

This theory is the most important mechanism in achieving adhesion. The most common surface forces that form at the adhesive-adherend interface are van der Waals forces. In addition, acid-base interactions and hydrogen bonds, generally considered a type of acid-base interaction, may also contribute to intrinsic adhesion forces. Research

has experimentally demonstrated that the mechanism of adhesion in many adhesive joints only involves interfacial secondary forces. The calculated attractive forces between two surfaces are considerably higher than the experimentally measured strength of adhesive joints; this discrepancy between theoretical and experimental strength values has been attributed to voids, defects or other geometric irregularities which may cause stress concentrations during loading.

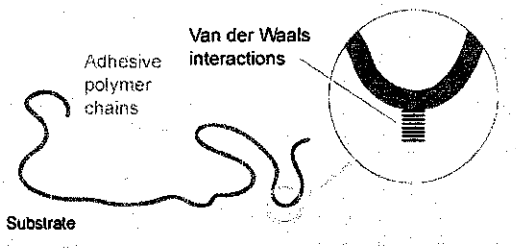


Figure 5: theory adsorption illustrations

To obtain good adsorption, intimate contact must be reached such that van der Waals interaction or the acid-base interaction or both take place; hence good wetting is essential. According to Young's equation, the surface tensions (liquid/vapor: γ_{LV}, solid/liquid: γ_{SL} and solid/vapor: γ_{SV}) at the three phase contacts are related to the equilibrium contact angle σ through:

$$\gamma_{SV} = \gamma_{SL} + \gamma_{LV} \cdot \cos \sigma$$

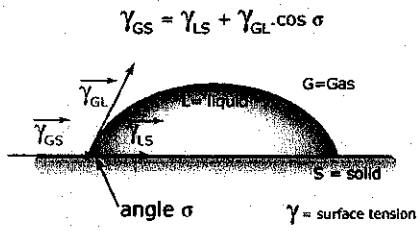


Figure 6: Wetting condition

The one important factor that influences the adhesive joint strength is the ability of the adhesive to spread spontaneously on the substrate when the joint is initially formed. For spontaneous wetting to occur:

$$\gamma_{SV} >= \gamma_{SL} + \gamma_{LV}$$

We can say that for good wetting: $\gamma_{SV} < \gamma_{LV}$

2.5.2 Chemisorptions Theory

The chemical bonding mechanism suggests that primary chemical bonds may form across the interface. Chemical bonds are strong and make a significant contribution to the intrinsic adhesion in some cases.

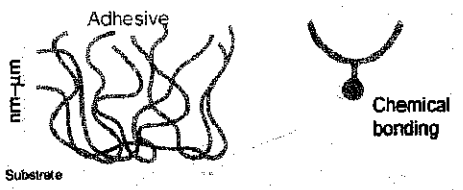


Figure 7: chemical bonding illustration

2.5.3 Mechanical Interlocking Theory

The mechanical interlocking theory of adhesion states that good adhesion occurs only when an adhesive penetrates into the pores, holes and crevices and other irregularities of the adhered surface of a substrate, and locks mechanically to the substrate. The adhesive must not only wet the substrate, but also have the right rheological properties to penetrate pores and openings in a reasonable time.

This theory explains a few examples adhesion such as rubber bonding to textiles and paper. Since good adhesion can occur between smooth adherend surfaces as well, it is clear that while interlocking helps promote adhesion, it is not really a generally applicable adhesion mechanism.

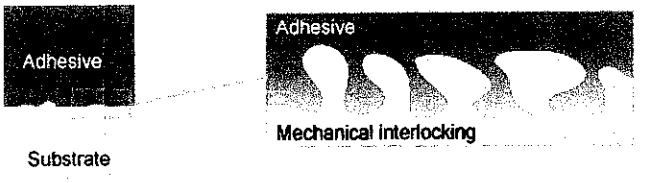


Figure 8: Mechanical interlocking theory illustration

2.5.4 Diffusion theory

The diffusion theory attributes the adhesion of polymeric materials to the inter-penetration of chains at the interface.

The major driving force for polymer autohesion and heterohesion is due to mutual diffusion of polymer molecules across the interface. This theory requires that both the adhesive and adherend are polymers, which are capable of movement and are mutually compatible and miscible.

Parameters affecting the diffusion process are: contact time, temperature, molecular weight of polymers and physical form (liquid, solid). Polarity generally increases adhesion.

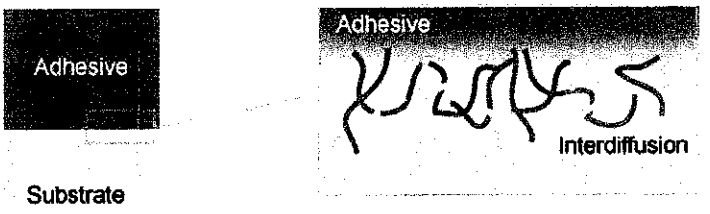


Figure 9: diffusion theory illustration.

The diffusion theory, however, has found limited application where the polymer and adherend are not soluble or the chain movement of the polymer is constrained by its highly crosslinked, crystalline structure, or when it is below its glass transition temperature.

2.5.5 Fracture Theory

The area of interfacial bonding between coating and substrate will, in most instances, contain voids or defects. The result is deviation between the ideal adhesive strength and the practical limit.

2.5.6 Weak Boundary Layer Theory

This theory maintains that true interracial fracture does not occur, and that fracture usually occurs cohesively in a weak boundary layer (WBL), which may be near the interface between coating and substrate. For a strong bond, the boundary layer (layers) must be rheologically sound and chemically durable.

2.5.7 Wetting-Contact Theory

The theory states that van der Waals attractive forces alone provide sufficient coating/substrate bond strength given perfect molecular contact, and that the extent of contact and resulting bond strength are functions of wetting energetics [13-14]. The lower the contact angles, the more the interracial area of contact, which generally yields improved adhesion.

CHAPTER 3
METHODOLOGY

3.1 PROJECT PLANNING AND GUIDELINES

The project was planned into three main phases. The initial phase was held on the beginning of 6 months as project started, and mainly focused on preparations, background studies, and concept studies. In the end of the first phase, the interim report was produced to give a rough idea of the project scope.

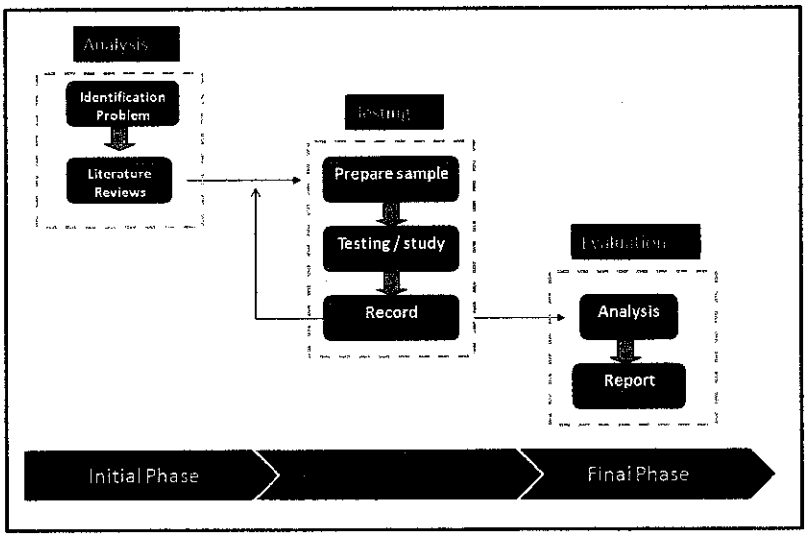


Figure 10: Flow chart of overall project

The intermediate phase also known as experiment and evaluation phase was carried on the next 4 months after initial phase completed, and covered all necessary

experiment and analysis. The results obtained from this phase was recorded and analyzed further during the final phase of the project.

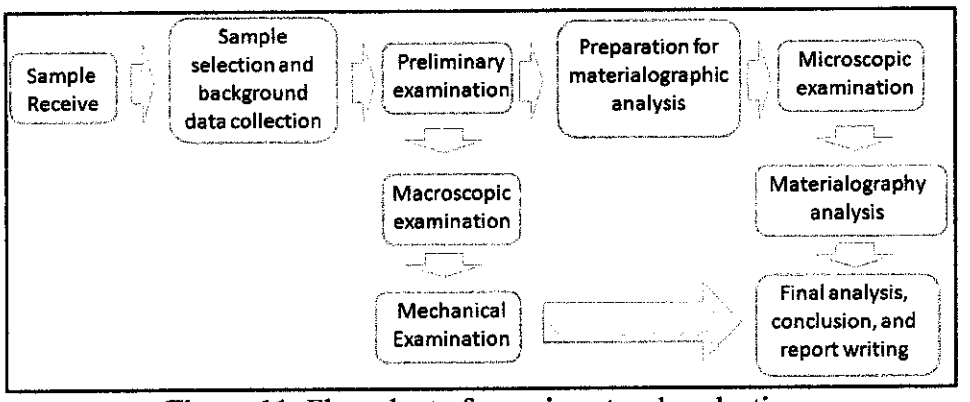


Figure 11: Flow chart of experiment and evaluation

A final report was produced on the final stage of the project by summarize all the result and explained project scope and the methodology in details.

3.2 TOOLS AND EQUIPMENTS

The tools and equipments involved in this project were as follows:

1. Abrasive cutter – use for specimen preparation;
2. Bed saw– use for specimen preparation;
3. Grinding Machine – use for specimen preparation;
4. Polishing Machine – use for specimen preparation;
5. Optical Microscope- use for microscopic analysis;
6. Micro Hardness – use for mechanical analysis; and
7. Scanning Electron Microscope (SEM) – use for materialography analysis.

3.3 PROCEDURE IDENTIFICATION

3.3.1 Background study

Background studies were done to collects the relevant data on intumescent material and previous researches work for project fundamental basic. All of these data were compared with results obtained from the experiment and examination in order to produce the good analysis result.

3.3.2 Sample Selection and Background data collection

Material

"Firetex M90 Epoxy intumescent" was the intumescent material used in this project supplied by the Normax (m) Sdn Bhd, one of intumescent coating manufacturer. The material was design for the oil and gas industries application with capability to withstand the high temperature of fire especially for hydrocarbon fire. While, the primer used in this project was "Epigrip L425HS Zinc Phosphate Primer" as the corrosion protection and enhancement bonding between intumescent and steel substrate. The details components for each coating layer used on the project were as Table 3.

TABLE 3: Sample coating material.

COATING LAYER*	MATERIAL
1st layer (Primer)	Epigrip L425HS Zinc Phosphate Primer – Light Grey
2nd Layer (Intumescent)	Firetex M90 Epoxy PFP
3rd Layer (Wire Mesh)	Firetex J120 Scrimcloth
4th Layer (Intumescent)	Firetex M90 Epoxy PFP
5th Layer (Top Coat)	Resistex C137V2 Special Firish - Holly 14C39

**the layer number increase as further from steel substrate*

Coating process practiced

The coating process practice for this project was the normal practical coating practiced on the industries. These coating processes were divided into 4 coating layer (Figure 12) and each layer was coated by using sprayed method. The process started with the surface preparation which included surface cleaning by sand blasting.

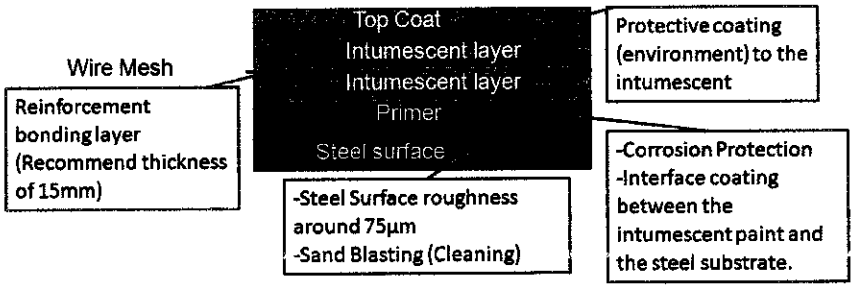


Figure 12: Coating sequences and descriptions.

The spray was directed in a back and forth motion across the surfaces of steel for about 4mm to 6mm thickness of single coating and the next coatings was applied after the prior coating was partially cured (for 30 minutes to 4 hours). The wire mesh was introduced in between 15mm of coating thickness as joint reinforcement inside the coating.

3.3.3 Sampling

Standard Operating Procedure

The standard operating procedures (SOPs) practiced in this project as follows:

1. The sample was prepared at least a day before the experiment.
2. Each coating layer was applied on the steel plate by using a spray, and each layer was applied in sequences after it dry.

3. Each layer was dried under the room condition without using any dryer which affect the quality of bonding.
4. Only good coating layer with no or minimum air bubble interfere were accepted as the sample of the project.
5. The sample was cut into small pieces according to the experiment requirement.
6. The samples used for cross-sectional analysis were cut by using the steel cutting machine and the samples with good cutting edge were accepted as a sample.
7. Each sample that used on the experiment was labeled according to date, number of layer, and layer composition.
8. The samples were stored on the storage box to prevent it from scratch, heat, and also water which can damage the sample.

Sample

The samples involved in this project were classified as:

1) **Normal Sample:** Intumescent coating without expose to jet fire.

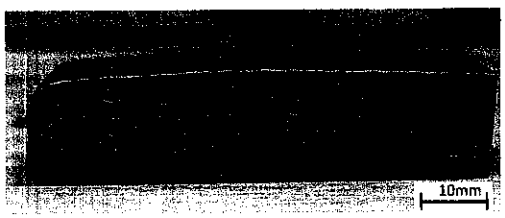


Figure 13: Normal sample

2) **Burned Sample:** Intumescent coating after expose to jet fire on top surface.

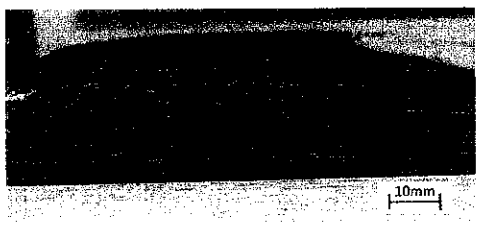


Figure 14: Burned Sample

The samples used in these project were undergo the same coating procedure which explain earlier (Table 3), by applied the coating layer on the big size steel plate and divided it into two section. One of these section' top layers (Burned sample) was heated by jet fire with the LPG (acetylene, C_2H_2) fire at 3000°C for 25 minutes, while another (normal sample) keep as its original condition without exposed to heat.

3.3.4 Sample preparation methodology for Microscopic Analysis

The samples preparations procedure for the microscopic analysis was follows the ASTM E3 Standard Practice for preparation procedure of the Metallographic Specimen.

By refer to the standard practice ASTM E3 and collective data from ASM guide book in focusing on the Metallographic and Microstructure of Low-Carbon and Coated Steel, the samples were undergo sectioning, mounting, grinding, polishing and etching process.

i. Sectioning process

Cutting process for preparing the specimen was divided into two stages which started with a rough cutting process using band saw machine with automatic medium feed rate, then follow by smooth cutting process using abrasive cutting machine.

The final dimensions of the sample received from the cutting process are in range of 1cm x 1cm to 2cm x 2cm.

ii. Cleaning

After the retrieved the sample with suitable size for mounting mould, the samples were clean from debris and other foreign material which may affect the analysis result, by using soft cleaning brush.

iii. Mounting

The samples were mounted by using cold mounting process. Cold mounting solution with1 ratio of hardener and 5 ratio of resin were poured into the mold and leave to solidify under the room condition for 24 hours.

Figure 15: Sample after mounted, on picture normal sample is on right side and Burned sample on the left side.

iv. Grinding & Polishing

The next stage for preparing the sample was grinding and polishing process. These processes were done on same day together with the etching process in order to prevent the sample from corrosion. A few data was gathered from ASM data book before these procedures were commenced such as the grinding papers grit sizes, polishing surface and etchants.

The grinding processes were divided into two stages, rough and fine grinding. These processes were conducted by the semi-automatic methods, by holding the sample manually on the automatic spinning grinding paper. The sequences of grinding process practice for this project were 120,240, 600, 800 and 1200 grit size. In each grit size, the specimen was rotated by 90° degree angle until the scratch produce by the grinding process were in one direction.

After the grinding process, the specimen was been cleaned thoroughly by water to remove the debris produce by the previous grinding process before proceed to polishing process. The polishing process start from the 6μm graded diamond paste and final polish

by 1μm diamond abrasive. The polishing process use in this project was semi-automated process, by holding the sample on rotating discs covered with soft cloth impregnated with micro-particles of diamond. This step was kept to the shortest time possible to prevent edge rounding, pitting, or other artifact.

Figure 16: Grinding and polishing process.

The polishing step was stopped after the metallic surface (low carbon steel) totally smooth by produced a scratch free mirror-like surface.

v. Etching

The etchants use for this project sample was 2% Nital by mixing 2% of nitric acid on the 98% ethanol.

3.3.5 Mechanical Examination: Micro Hardness

The mechanical examination conducted for this project was micro hardness test with Vickers hardness. The area subjected to hardness examination was on cross-section of carbon steel with fixed average distance of 0.5mm from the carbon steel plate surface with primer (Figure 17).

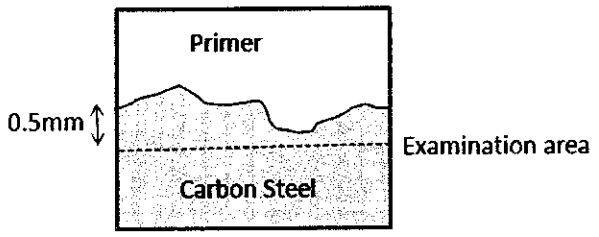


Figure 17: Hardness examination area

The testing was repeated 20 times for each sample with the same latitudinal distance from the carbon steel surface.

3.3.6 Microscopic analysis

Microscopic analysis was carried out by optical microscope. The mounted samples were analyzed from the lowest magnification increased until the appropriate level microstructure was observed. The scale marker for the sample was obtained by measure the Vickers hardness mark length at the same magnification level of sample.

3.3.7 Materialographic analysis

The Materialographic analysis was conducted by the Scanning Electron Microscope (SEM) machine with 3 type analysis; SEM imaging, SEM QBSD (four-quadrant back-scattered detector), SEM EDS (Energy Dispersive X-Ray Spectrometer).

The specimen were coated by the conductor particles as a requirement to be able the specimen to conduct the electron pass through it.

3.3.8 Image area estimation

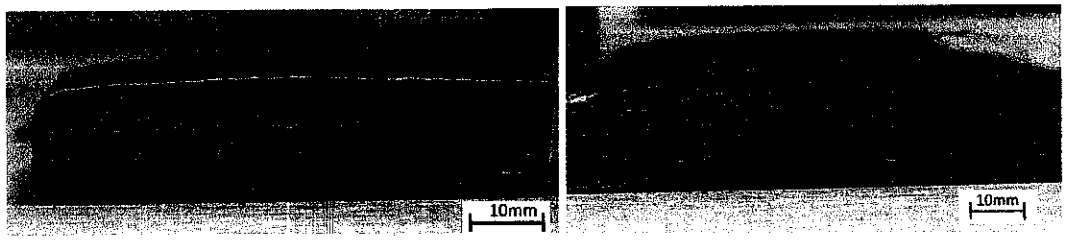
The overall of this project methodology was mainly based on the observation and observer's judgment, which make a calculation data is crucially needed to support the evaluation for a proved, such as the value of distance and area calculation from the photo obtains on the both Macroscopic and Microscopic analysis. The 5mm x 5mm small boxes were manually draw on the A4 printed photo with scale as the ruler in estimating and calculating the distance or area of pore spaces, effected area, and coating on the photo. This method was adapted for this project due to no image analysis software is available and this conventional method used widely in determining the area on photo.

CHAPTER 4

RESULT AND DISCUSSION

4.1 INTUMESCENT COMPONENTS REACTIONS

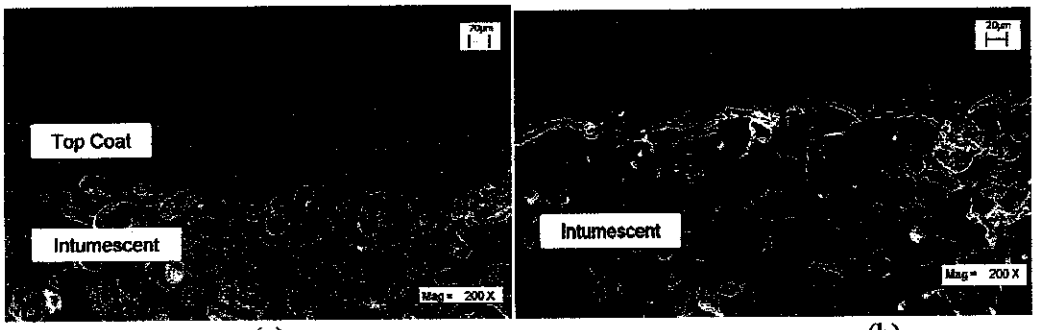
About 3000°C heat temperature from the hydrocarbon sources fire were subjected to the intumescent coating during sample preparation of Burned Sample, enough to melt the entire low carbon steel plate in few minutes. Refers to results obtain from project, the reaction of intumescent were simulated as started with the degradation of the top coat, the environment protection layer for coating. This degradation process was observed on the Burned Sample (Figure 18, 19) where the only top layer of intumescent was observed on top of coating layer. While, the normal sample still retain its top coat with the same condition as it prepared.



(a) (b)

Figure 18: Macroscopic photograph of cross-section coating layer (a) Normal Sample. (b) Burned Sample.

During the degradation of top coat, the heat generated from fire start to spread towards the top layer of intumescent coating, and epoxy resin start to soften. As the heats received by the intumescent layer increasing to certain level, blowing agent which was Calcium Carbonate, $CaCO_3$ (Table 4) released carbon dioxide at a temperature at which cured epoxy resin is soft but which is below the temperature at which carbonaceous char is formed.



(a) (b)

Figure 19: SEM photograph of top side of the (a) Normal Sample. 200X, (b) Burned Sample. 200X.

TABLE 4: Summarize Chemical analysis of intumescent layer.

COMPONENT	ELEMENT
Blowing agent	$CaCO_3$
Fire retardants	KCl (halogen fire retardants) GaP (char forming agent)
Filler	SiO_3
Fluxing agent	Ti

The char which was formed expanded and thereby better insulates the substrate as expansion gas was retained within the char. Result from these reactions were the shrinkage of micro pore spaces which were observed on the Burned Sample (Figure 19). The shrinkage of micro pores were occurred when the gas inside the pore (Blowing agent) were released (Figure 20).

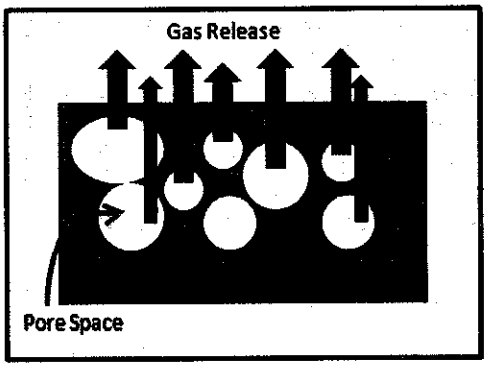


Figure 20: Reaction of blowing agent towards heat.

The shrinkage were observed after compared the SEM photograph for the both sample, where the average size of $3.6x10^{-10}$ m^2 pore space were seen on the top layer of Normal Sample coating. While, average size of $4.3x10^{-11}$ m^2 pore space were observed on the Burned Sample (Figure 19).

TABLE 5: EDS result on the intumescent layer on random position

ELEMENT	WEIGHT %	ATOMIC %
C	45.50	55.86
O	43.21	39.83
Mg	0.72	0.44
Si	0.67	0.35
P	1.07	0.51
Cl	0.30	0.12
Ca	6.01	2.21
Ti	0.90	0.28
Fe	0.47	0.12
Cu	1.14	0.27
Totals	100.00	100.00

During the heating process, two fire retardant elements which are chlorinated paraffin from KCl (Table 4) as halogen fire retardants and Phosphorous elements inside GaP (Table 4) as char forming agent were reacts as intumescent catalyst [22] and promotes

the formation of char when intumescent coating was exposed to fire. These two importance elements were acts as fire retardant by control the rate and mechanism of thermal decomposition of cured epoxy, results in formation of carbonaceous char instead of carbon monoxide, carbon dioxide and water.

The filler element which refers to the ceramic material, SiO_3 (Table 4) inside the coating, reduce the intumescent coating density and also reduce thermal conductivity of coating and of its resultant char.

A part from this reaction, fluxing agent which identify as the Titanium, Ti elements from EDS result (Table 5), react with at least a portion of the phosphorous coating component and at least a portion of the silicon containing component to form an at least partially softened or liquid, phosphorosilicate glass which expands and form within the char due to gases released by thermal decomposition of one or more components of the intumescent coating. These formations of insulated char continued to growth and destroyed until the entire intumescent coatings were reacted to the heat.

4.2 MICRO SIZE PORE

On the microscopic analysis, a group of empty spaces (pore) were observed inside the coating layer especially on intumecsent layer and primer with the average size of $4.3x10^{-11}$ to $3.6x10^{-10}$ m^2 (Figure 19). These micro size pores were considered as optimum size at which it not affects the overall coating bonding due to the size was too small to weaken the contact boundaries. While the macro size pores were observed by naked eyes on both samples weaken the bonding performance (Figure 18). The macroscopic photograph obtains (Figure18) revealed about 1.2% ($10mm^2$) of $902mm^2$ coating area of Normal Sample, and for the Burned sample, about 0.9% ($15.8mm^2$) of the total $1742mm^2$ coating area full fill by the pore space with the $7.5mm^2$ of the biggest single pore area detected (Figure 21). These abnormal were affected by wind interfered during the coating applied.

The micro size pore was not accidently produced during coating but actually be designed for the low density coating, by minimize the coating volume without drag the coating performance down, and typically these empty spaces were accommodated by the blowing agent in form of gas. To sufficiently insulate component against fire, large masses of coating must be applied which significantly increase the weight load on the coated component. This increased weight load imposes structural and economic liabilities [21].

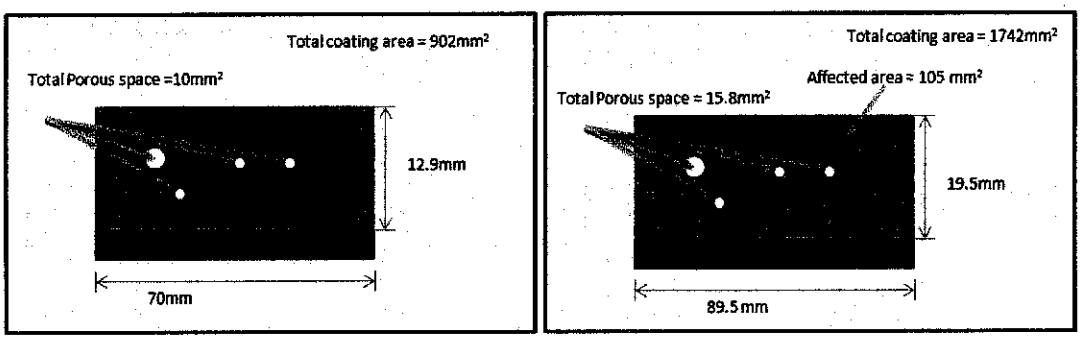


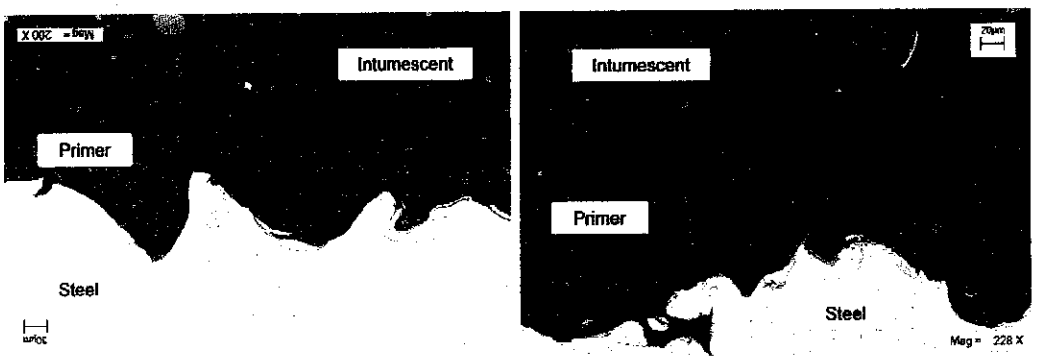
Figure 21: Summary of image analysis result on cross-section of (a) Normal Sample, (b) Burned Sample.

By observing and comparing microscopic photograph of both sample, the pore space shrinkage can be clearly observed on the top layer of Burned Sample, due to the blowing agent which accommodate on pore space reacts towards the heat by releasing the carbon dioxide. The average size of $3.6x10^{-10}$ m^2 pore space were appears on the top layer of Normal Sample coating. While, average size of $4.3x10^{-11}$ m^2 pore space were observed on the Burned Sample (Figure 19). As the reaction continues, the pore spaces degrade and shrink as the carbon dioxide inside the blowing agent leaving the space. This reaction was the one key indicator for indentify the heat present on the area.

4.3 COATING BONDING

Coating bonding is another indicator to determine the quality of the coating performance and estimate the durability of the coating; and any changes of these aspects before and after heated were used for measurement of the coating ability as the fire resistive coating. There are two types of bonding were observed in this project which is the mechanical interlocking and adhesive bonding. On the certain area, there will be either mechanical interlocking bonding or adhesive bonding or both involve on the same area.

On the top layer of Normal Sample, the bonding interface between the top coat and intumescent coating shown the only adhesive bonding were occur on this region; indicated by the well bounded between two different materials without a gaps (Figure 19.a). The good wetting condition was clearly observed on the contact region between the top coat and the intumescent.



(a) (b)

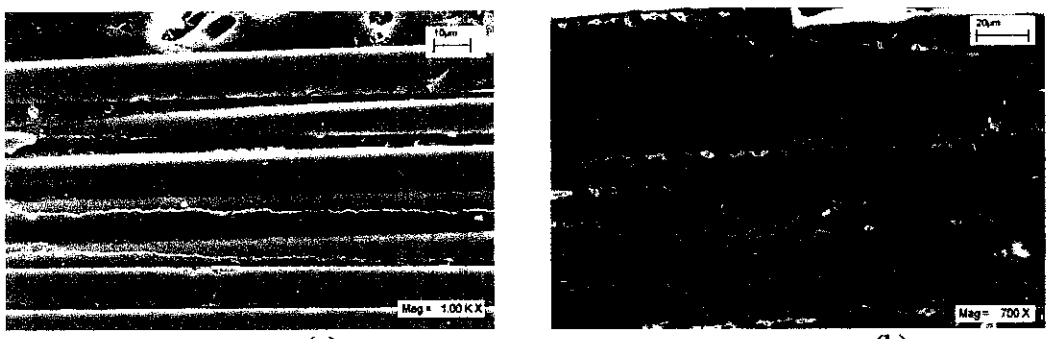
Figure 22: SEM with QBSD photograph of carbon steel, primer and intumescent of (a) Normal Sample. 200X, (b) Burned Sample. 228X.

While on the Burned Sample's top layer, no visible top coat was observed above the intumescent layer. The top coat was destroyed due to the heat generated from fire, and indicates the top coat had poor fire resistivity coating characteristic. After the coating exposed to the heat, it lost its protection layer permanently and need to be

recoated for reactivate the environment protection back, which was not provided by the intumescent layer it's self.

As the analysis continues further down to the border of the contact region with primer, the analysis revealed the adhesive bonding was occurred on the both sample without a single gaps (Figure 22a). The wetting condition between the both materials were identified as good as refers to well bounded of two difference composition material together as one (Figure 5). After being heated by the fire on the coating surface, the bonding between the intumescent and primer was unaffected with no gaps or slip can be observed throughout this region (Figure 22b).

For thicker coating, the reinforcement bonding like carbon fiber mesh need to be introduced for the bonding strengthened and hold up the coating together. In this work, the arrangement of the carbon fiber mesh were observed provides a good mechanical interlocking for reinforcement of the coating, and increase the coating durability by preventing the coating from slipping or wear off during the reaction due to the weak coating bonding. Typically, the carbon fiber mesh was implemented into the coating on every 15mm of overall coating thickness regardless the coating sequences.

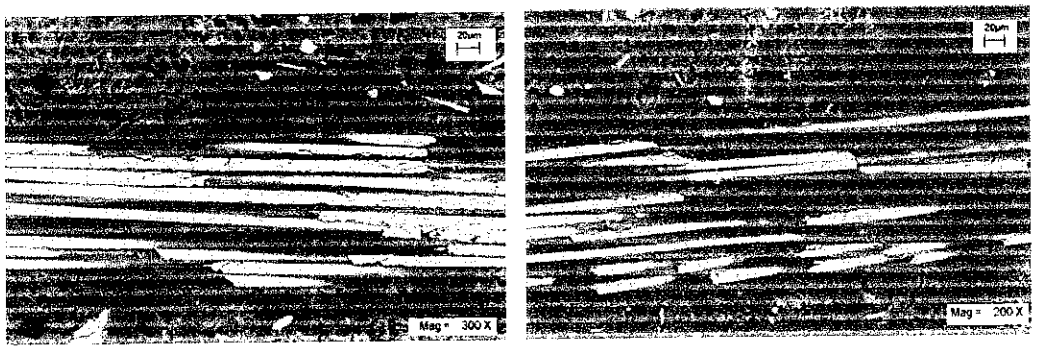


(a) (b)

Figure 23: SEM photograph of carbon fiber mesh of the (a) Normal Sample. 1000X, (b) Burned Sample, 700X.

As for the both sample photographs (Figure 23 and 24), on the carbon fiber mesh bonding region shown a good bonding and not much differences was clearly being

observed on this region beside the shrinkage of the pore spaces on the intumescent layer over the carbon fiber mesh on the Burned Sample (Figure 23.b). The carbon fiber mesh arrangement and the contacting with the intumescent were relatively unaffected on the Burned Sample.



(a) (b)

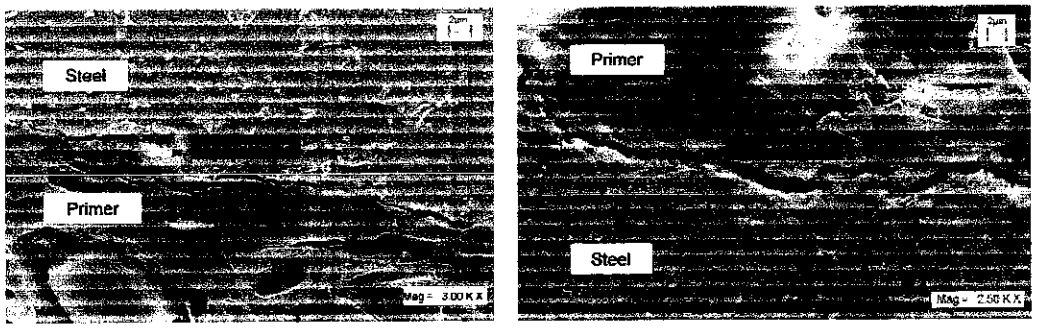
Figure 24: SEM with QBSD photograph of carbon fiber mesh of (a) Normal Sample. 300X, (b) Burned Sample. 200X.

The first coating layer or the based coat of this coating project was primer. The chemical analysis (Table 6) on primer reveals the Zinc (Zn) and Phosphorus (P) elements were exist on the primer solution, and proved the primer materials were zinc phosphate as claim by the supplier. The zinc is very importance element in primer as corrosion protection agent for the carbon steel.

TABLE 6: Primer chemical contents

ELEMENT	WEIGHT %	ATOMIC %
O	46.98	65.15
Mg	12.13	11.07
Al	1.15	0.95
Si	13.81	10.91
P	3.02	2.16
Ca	3.85	2.13
Ti	8.46	3.92
Fe	2.02	0.80
Zn	8.57	2.91
Totals	100.00	100.00

Before the primer was applied on the steel surface, few preparations were being done in order to provide a good bonding region and fulfill the sub-coating layer purpose. The mechanical interlocking and week adhesive bonding were observed on the contact region between the steel substrate and the primer. Around these regions, the SEM photograph of both samples shown several gaps were existed between the primer and the substrate (Figure 25), and the primer applied as the corrosion protection was for nothing.



(a) (b)

Figure 25: SEM photograph gap between primer and carbon steel of the (a) Normal Sample. 3000X, (b) BurnedSample. 2500X.

The durability and effectiveness of zinc phosphate primer as the corrosion prevention layer was reduced by these gaps and also reduce the overall coating strength between the overall coating layers to substrate. These gaps occurs due to the surface roughness of the steel substrate, prevented a partial of primer from filled the gaps and stick to the steel surface and also the low wetting condition between both materials (Figure 26).

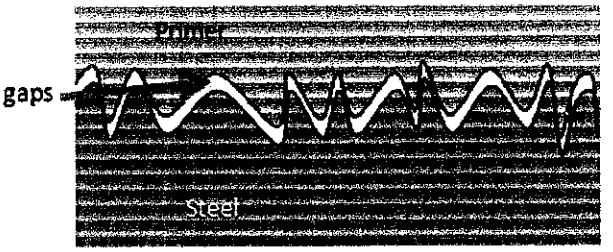
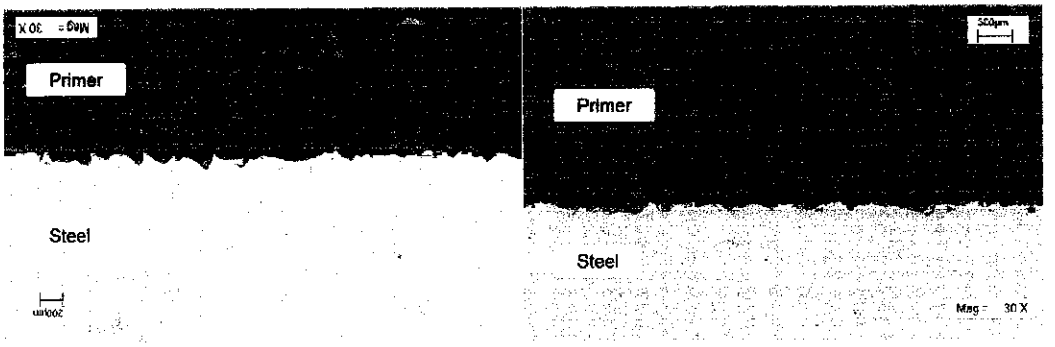


Figure 26: The surface roughness prevent the primer to adhere with steel substrate

After compared the both samples SEM photographs, the gaps were expanded or growth bigger as responded to heat supplied to the coating surface (Figure 25). The random measured gap thickness of Burned Sample was 1.4μm and 607.7nm for Normal Sample. These gaps expanded were due to the small expansion of the steel substrate responded to the small heat received. Even though the heat was being proved transfer inside the coating layer by expansion of the substrate and the pore size shrinkage, the amount of this heat was too small to change the overall steel microstructure inside the coating even though the coating was exposed to "steel melting temperature" heat. These coating not filtered 100% of heat from spreads inside the coating.



(a) (b)

Figure 27: SEM with QBSD photograph of carbon steel and primer of (a) Normal Sample. 30X, (b) BurnedSample.30X.

Although the adhesive bonding was not fully developed in this region, the mechanical interlocking was clearly observed in this region. The SEM with QBSD photographs of both samples (Figure 25 and 27) reveals the surface profiles of carbon steel were good for mechanical interlocking to occur on the region. The mechanical interlocking is good bonding mechanism especially between the low wetting contact materials, by one material lock the another material.

4.4 HEAT PENETRATION PERFORMANCE

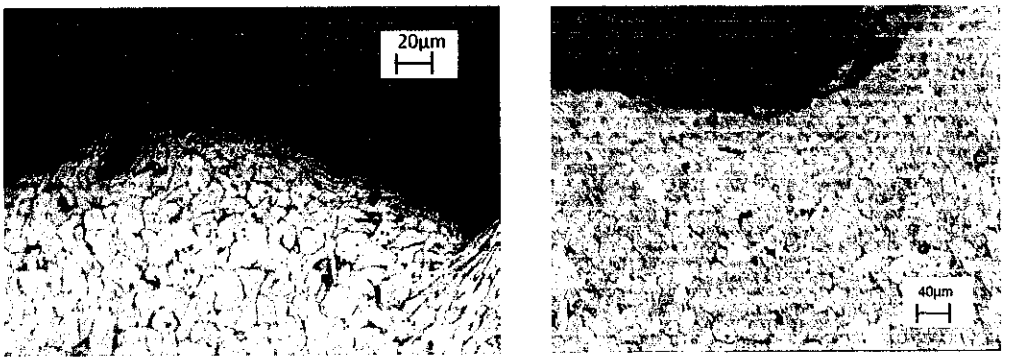
The macroscopic photograph of the Burned Sample shown only the upper layer of intumescent was affected by the heat (Figure 18.b). These affected layers only consumed 6% (105 mm^2) of the total 1742mm^2 volume of cross-sectional coating (Figure 21), and proved the coating can sustain the heat for longer time duration. Beside this, the photograph also not reveals any visible evident that can indicate the heat has been spread to steel substrate, and also shown the other layer below still not intact by the heat.

TABLE 7: Vickers Hardness, (HV) result

NO	NORMAL SAMPLE (HV)	BURNED SAMPLE (HV)
1	105.3	107.6
2	106.4	94.2
3	105.9	106.9
4	105.7	97.2
5	104.5	104.4
6	104.5	109.5
7	110.2	104.8
8	102.4	102.3
9	100.3	100.5
10	106.2	105.6
11	104.4	107.5
12	108.0	105.6
13	105.6	104.8
14	104.7	100.9
15	103.5	105.4
16	105.2	107.2
17	104.6	106.3
18	104.5	104.5
19	105.1	105.6
20	105.3	100.0
Average	105.115	104.04

Microhardness (Vickers, HV) examination under 300gf loads on the carbon steel plate revealed the steel physical characteristic in terms of hardness value. The testing was focused on the carbon steel surface 0.5mm below the steel substrate. Two samples were labeled as normal sample (without heated) and Burned sample (exposed to jet fire) with 20 separate test each. The values obtain were compared with each other in order to determine the microstructure changes of the steel under difference situation. The averages values of the two samples indicate the hardness values of the steel plate are almost unchanged with small differences of 1.075 HV (Table 7).

This result proved the ability of the coating as fire resistive coating for the steel plate, by preventing the heat from spreads to the steel substrate which may result the microstructure changes of steel. This small differences can be ignored because of the errors during the value reading and computation, or the compaction during the applying the coating layer.



(a) (b)

Figure 28: Optical microscope photograph of steel substrate with 2% Nital as etching agent of the (a) Normal Sample. 200X, (b) Burned Sample. 100X.

Meanwhile, the microscopic analysis via optical microscope was conducted to investigate the steel microstructure changes due to coating and heat. Across the carbon steel microstructure (Figure 28), a layer of microstructure deformation was appears on the top layer of the carbon steel substrate on the both sample. These deformations were

due to the sand blasting process during the coating preparation, the deformation was only observed on the area near the steel surface with distance of 15μm and steel grain size was back to normal size below this level. This deformation characteristic was not affected by heat from jet fire or hot rolling process. These surface roughness of steel was appears to be a good mechanical interlocking for the coating.

CHAPTER 5

CONCLUSION

5.1 CONCLUSION

The results gathers from this work had demonstrated the intumescent coating used was a good coating practiced as fire protection system and the steel integrity was observed untouched by fire. The conclusion for the coating performance as follows:

- The hardness value of the carbon steel was unchanged after the coating exposed to the jet fire; and
- No reaction was observed on the intumescent material on the area near the steel substrate by compared the size and concentration of pore inside the intumescent material.

The results also address the mechanical interlocking and adhesive bondings were the two types of bonding found inside the coating. The coating bonding and integrity summarize as follows:

- The mechanical interlocking was observed on bonding between primer and carbon steel substrate for both samples;
- Adhesive bonding was revealed inside the intumescent coating layers on area between
    - primer and intumescent;
    - intumescent and intumescent layer ; and
    - intumescent and top coat.

- And, the wire mesh implanted inside the coating layer shown a good mechanical interlocking with intumescent layer.

In summary, the studies of the interface coating are very importance in order to enhance the ability of intumescents as fire-retardant material such as the efficiency of coating bonding.

5.2 RECOMMENDATION

According to the results obtained earlier, performance of primer as the corrosion protection was limited or decreased by the gaps found between the primer and steel substrate. On the gaps area, the steel substrates were not covered by the primer. The corrosion will be initiated on this region if the gaps were filled by the water vapor. The improvement need to be done on this problem by eliminated the gaps. The gaps can be eliminated by produce a good surface roughness of the steel and used primer with high wetting characteristic.

REFERENCES

[1] "Ecuador oil pipe blast kills six", BBC News Network, *http://news.bbc.co.uk/1/hi/world/americas/1069005.stm* , 10 April 2008, 12.30AM.

[2] "Oil Pipeline Fire in Minnestota Kills 2 workers", 29 November 2007, Fox News Network
http://www.foxnews.com/story/0,2933,313688,00.html , 10 April 2008, 12.33AM

[3] J.A. Rhys, "Fire and Materials Volume 4 Issue 3". John Wiley & Sons, Ltd, 2004 (pp. 154-156).

[4] Slysh, Roman, "J Paint Technol Volume 47 Issue 607", Elsevier BV, August 1975, (pp 31-36)

[5] G. Camino, G. Martinasso and L. Costa, "Polymer Degradation and Stability Volume 27 Issue 3", Elsevier BV, 1990, (pp 285-296)

[6] R. Delobel, M. Le Bras, N. Ouassou and F. Alistiqsa, "Journal of Fire Sciences Volume 8 No. 2", 1990, SAGE Publications (pp 85-108).

[7] M. Jimenez, S. Duquesne and S. Bourbigot, "Thermochimica Acta Volume 449 Issues 1-2", Elsevier B.V., 1 October 2006, (pp 17)

[8] "Intumescent - Wikipedia, the free encyclopedia", http://en.wikipedia.org/wiki/Intumescent, 18 Feb 2008, 10.02PM

[9] Y. Sakumoto, J. Nagata, A. Kodaira, and Y. Saito (2001), "Durability Evaluation of Intumescent Coating for Steel Frames" (pp. 274)

[10] G.K. Castle, "Loss Prevention Volume 8", Elsevier BV, 1974, (pp 57-64).

[11] http://www.nullifire.com, 20 April 2008, 2.32 PM

[12] Mittal, K. L., "Adhesion Measurement of Thin Films, Thick Films, and Bulk Coatings, ASTM STP 640", American Society for Testing and Materials (ASTM), Philadelphia, PA, 1978, (pp 7-8)

[13] Sharpe, L. H. and Schonhorn, H., "Advances on Chemistry Volume 43", 1964, (pp. 189).

[14] Smarook, W. H. and Bonotto, S., "Polymer Engineering and Science Volume 8", 1968, (pp. 41)

[15] M. Wladyka-Przybylak, R. Kozlowski, "Fire and Materials", John Wiley & Sons, Ltd, 1999, (pp. 33)
[16] G. Montaudo, E. Scamporino, D. Vitalini, "J. Polym. Sci. Polym. Chem", 1983, (pp. 3361).

[17] C. Siat, S. Bourbigot, M. Le Bras, in: M. Lewin (Ed.), "Recent Advances in Flame Retardancy of Polymeric Materials", Business Communications Co, Norwalk, 1997, (pp. 318–326).

[18] N.C. Billingham,M. Bugajny, M. LeBras, S. Bourbigot, R. Delobel, "Polymer Degradation And Stability" , Elsevier Ltd , 1999, (pp. 264 – 266)

[19] "Engineering fundamentals, general properties of carbon steels" http://www.efunda.com/materials/alloys/carbon_steels/carbon.cfm, 7 August 2008, 12AM

[20] "Engineering fundamentals, general properties of steel", http://www.efunda.com/materials/alloys/alloy_home/steels_properties.cfm, 7 August 2008, 1.05AM

[21] Joseph W. Hanafin, David C. Bertrand,"Low Density, Light Weight Intumescent Coating, 6096812", United States Patent, 1 August 2000.

[22] Michael John Sinclair, James Edward Watts, "Fire Retardant Intumescent Coating, US 7217753 B2", United States Patent, 12 May 2005.

APPENDIX I

PROJECT GANTT CHART

A) Gantt Chart (1st semester)

No	Details	Week													
		1	2	3	4	5	6	7		8	9	10	11	12	13	14
1	Project topic selection	■							MID SEMESTER BREAK							
2	Meeting with supervisor (AP Dr. Patthi Hussain)		■	■	■	■	■	■		■	■	■	■	■	■	
3	Preliminary research work															
	a) Studies the detail about intumescent material and steel pipes use in oil & gas industries			■	■	■	■									
	b) Studies on coating method (paint)					■	■	■								
4	Preliminary report preparation			■												
5	Submission of preliminary report (15th Feb 2008)				■											
6	Seminar 1					■	■	■								
7	Progress report preparation							■								
8	Submission of progress report									■						
9	Studies the suitable material for the experiment							■		■	■	■				
10	Studies for the experiment that need to be carry out									■	■	■				
11	Get familiar with the interface study & analysis											■	■	■		
12	Submission for interim report final draft														■	
13	Oral presentation preparation													■	■	
14	Oral presentation															■

B) Gantt Chart (2st semester)

No	Details	Week													
		1	2	3	4	5	6	7		8	9	10	11	12	13	14
1	Acquire the sample	■							MID SEMESTER BREAK							
2	Meeting with supervisor (AP Dr. Patthi Hussain)		■	■	■	■	■	■		■	■	■	■	■	■	
3	Preparation specimens															
	a) metallographic specimens		■	■	■											
	b) Hardness					■										
4	Testing /analysis															
	a) Macroscopic		■													
	b) Optical Microscopy				■											
	c) SEM					■										
	d) Hardness test						■									
5	Submission progress report				■											
6	Analysis / Compilation		■	■	■	■	■	■		■	■					
7	Seminar 1									■						
8	Poster exhibition											■				
9	Submission of project dissertation (soft-bound)													■		
10	Oral presentation														■	
11	Submission of project dissertation (hard-bound)															■

APPENDIX II

ENERGY DISPERSIVE X-RAY SPECTROMETER (EDS) RESULT

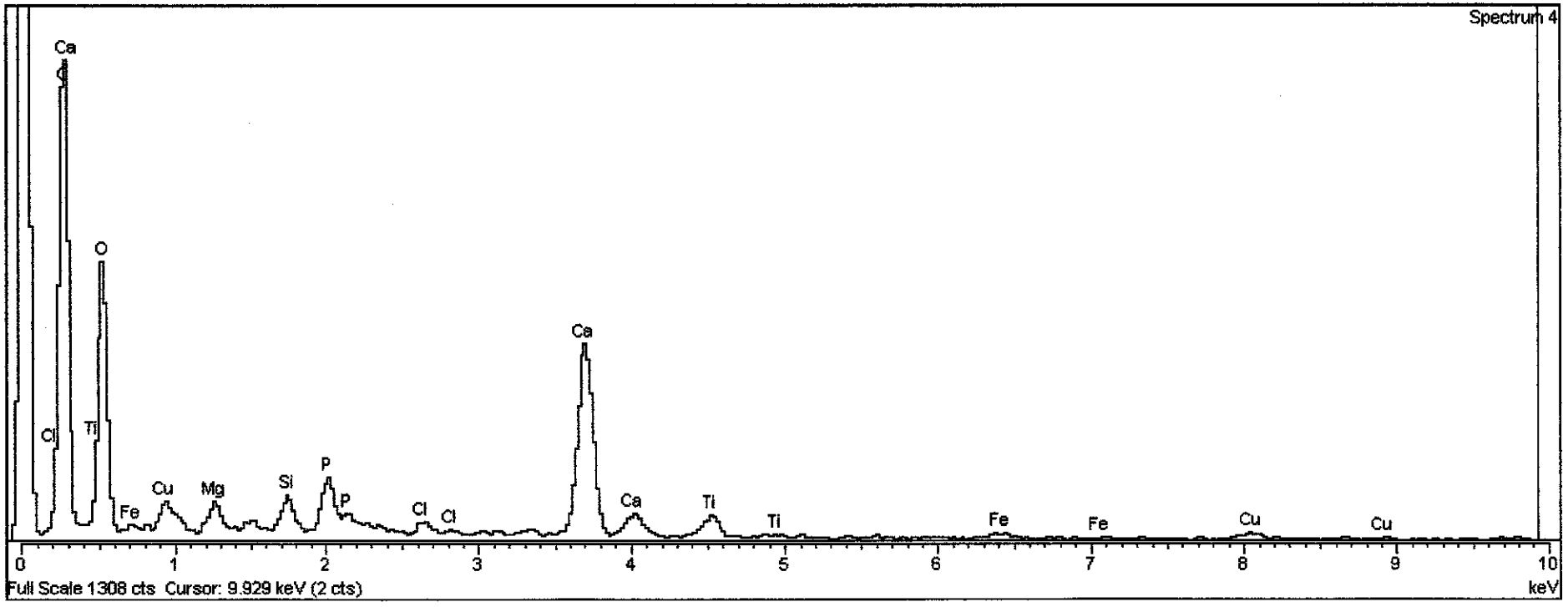
EDS Result # 1on Intumescent

Spectrum processing :
No peaks omitted

Processing option : All elements analyzed (Normalised)
Number of iterations = 5

Standard :
C CaCO3 1-Jun-1999 12:00 AM
O SiO2 1-Jun-1999 12:00 AM
Mg MgO 1-Jun-1999 12:00 AM
Si SiO2 1-Jun-1999 12:00 AM
P GaP 1-Jun-1999 12:00 AM
Cl KCl 1-Jun-1999 12:00 AM
Ca Wollastonite 1-Jun-1999 12:00 AM
Ti Ti 1-Jun-1999 12:00 AM
Fe Fe 1-Jun-1999 12:00 AM
Cu Cu 1-Jun-1999 12:00 AM

Element	Weight%	Atomic%
C K	45.50	55.86
O K	43.21	39.83
Mg K	0.72	0.44
Si K	0.67	0.35
P K	1.07	0.51
Cl K	0.30	0.12
Ca K	6.01	2.21
Ti K	0.90	0.28
Fe K	0.47	0.12
Cu K	1.14	0.27
Totals	100.00	



EDS Result #2 on Intumescent

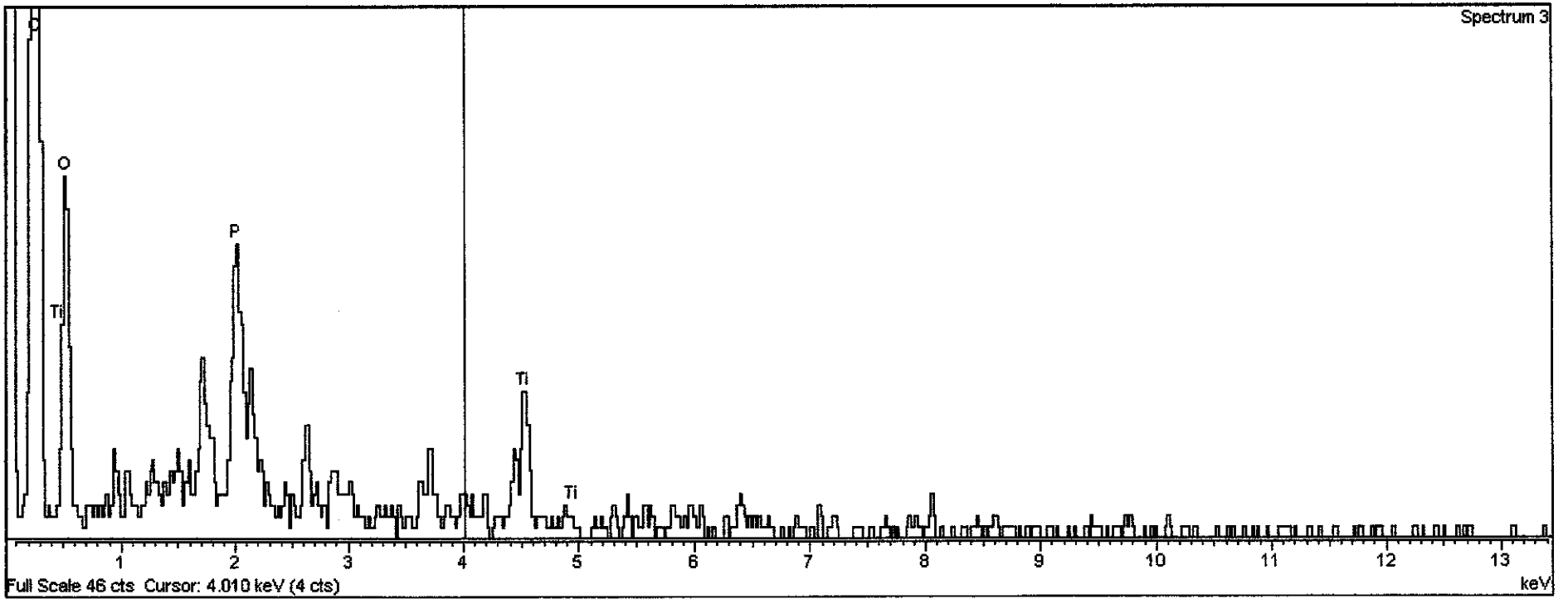
8/13/2008 4:05:24 PM

Spectrum processing :
No peaks omitted

Processing option : All elements analyzed (Normalised)
Number of iterations = 5

Standard :
C CaCO3 1-Jun-1999 12:00 AM
O SiO2 1-Jun-1999 12:00 AM
P GaP 1-Jun-1999 12:00 AM
Ti Ti 1-Jun-1999 12:00 AM

Element	Weight%	Atomic%
C K	66.38	74.03
O K	29.13	24.39
P K	2.13	0.92
Ti K	2.36	0.66
Totals	100.00	



EDS Result on Primer

Spectrum processing :
Peak possibly omitted : 2.850 keV

Processing option : All elements analyzed (Normalised)
Number of iterations = 5

Standard :
O SiO2 1-Jun-1999 12:00 AM
Mg MgO 1-Jun-1999 12:00 AM
Al Al2O3 1-Jun-1999 12:00 AM
Si SiO2 1-Jun-1999 12:00 AM
P GaP 1-Jun-1999 12:00 AM
Ca Wollastonite 1-Jun-1999 12:00 AM
Ti Ti 1-Jun-1999 12:00 AM
Fe Fe 1-Jun-1999 12:00 AM
Zn Zn 1-Jun-1999 12:00 AM

Element	Weight%	Atomic%
O K	46.98	65.15
Mg K	12.13	11.07
Al K	1.15	0.95
Si K	13.81	10.91
P K	3.02	2.16
Ca K	3.85	2.13
Ti K	8.46	3.92
Fe K	2.02	0.80
Zn K	8.57	2.91
Totals	100.00